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Electron Microscopy for the Pulp and Paper Industry

D.R. Rothbard

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INSTITUTE OF PAPER SCIENCE AND TECHNOLOGY
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Author: David R. Rothbard
Institute of Paper Science and Technology, Atlanta, Georgia

David R. Rothbard, Ph.D.
Associate Scientist
Manager, Microscopy and Fiber Analysis
Institute of Paper Science and Technology
500 10th Street, NW
Atlanta, GA 30318 USA
Phone 404-894-5742
Fax 404-894-4778
email: david.rothbard@ipst.edu

1. INTRODUCTION

Electron microscopy (EM) is one approach to the analysis of paper and related materials for problem solving, product development, and technology improvement in the paper industry. Most materials requiring EM evaluation will also have another form of microscopy, or physical or chemical testing performed. While EM is clearly the best method for some situations, there are other instances where it is simply preferred because a small amount of material is available, preparation is easier, or another method is not readily available. Digital image acquisition and the World Wide Web have accelerated the process of image acquisition and communication of EM analyses.

Both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have their applications in paper science. SEM is more commonly used in the paper industry than TEM due to the nature of paper industry needs and, perhaps, the ease of preparation and operation. SEM images of paper surfaces may reveal the degree of fiber bonding and the amount of external fibrillation. One can also look at the distribution of mineral fillers with backscattered electron (BSE) imaging and identify them using energy dispersive x-ray spectroscopy (EDS) analysis.

The SEM easily portrays differences in the microtexture of papers. Figures 1 and 2 compare facial tissue with xerographic paper. Notice the open, porous structure of the tissue and the crimping of fibers from creping, embossing, or folding. The xerographic paper is heavily loaded with calcium carbonate filler to optimize smoothness and reduce fiber usage.

By examining cross sections of paper and board with SEM, important z-dimensional properties can be measured. These include the density and porosity of the sheet, the thickness

and uniformity of coatings, and the retention of filler. Also, the composition and microstructure of coatings can be determined. Bonds between the fluted medium and liner in corrugated board have been evaluated by SEM. The collapse of individual fibers and, sometimes, their internal fibrillation can be revealed.

The raw materials of papermaking can be characterized by EM to improve their performance or for quality control in production. The study of raw materials might include the morphology of wood and plant cells. SEM is commonly applied to evaluation of pigments used in fillers and coatings. At higher magnifications, polymeric sizing agents and latex binders can be visualized by TEM.

Operational problems in pulp and paper mills are sometimes resolved with the aid of SEM. Scales in pipes or vessels can develop in the evaporators of a pulp mill or the bleach plant of a paper mill. Shutdowns for maintenance cause expensive interruptions in production. SEM (along with x-ray diffraction and light microscopy) is commonly used to determine the texture and composition of mill scales. With this information, mill operations can be modified to minimize scale formation and expedite removal. Understanding corrosion in mills sometimes requires the use of SEM/EDS or TEM.

Pulping liquors used in the kraft process may contain solids derived from feed chemicals, wood chips, or corrosion. Black liquor from the recovery boiler or green liquor from the washing of boiler residue contains fine solids that can be measured and chemically analyzed by SEM/EDS. The texture of fume particles that are produced in recovery boilers, as well as chars that are generated in black liquor gasification, have been measured by SEM to understand the effects of modifying control variables.

Contaminant particles, spots, and other variations that cause nonuniform appearance in products are investigated with a combined approach. This typically begins with light microscopy and may include FTIR or SEM/EDS. Sometimes the sources of these contaminants are the containers in which raw materials were packaged or transported. Off-specification paper that is repulped (known as broke) can pick up contaminants from the mill. Screening devices of various designs are generally effective in removing particulate contaminants from the pulp. However, deposits from components of virgin or recycled paper (e.g., fibers, stickies, fines, fillers) can build up on rolls and roll fabrics in the natural course of mill operation.

2. PULP AND PAPER PRODUCTION

2.1 Wood Fiber and Papermaking

Paper is normally formed from a water suspension of wood-derived cellulose fibers brought into very close contact after removal of excess water by drainage and web consolidation, in order to develop bonds. These are primarily hydrogen bonds whose formation is catalyzed by surface tension and mechanical forces. Individual fibers are liberated during pulping and refined to shorten, flatten, create fines, and to increase flexibility, hydration, and fibrillation. Refining and water removal improve the bonding of paper fibers. Exiting the headbox of a paper machine, the fiber suspension might contain 99% water. Under normal room conditions, finished paper typically has a 5 to 8% water (moisture) content.

Wood fiber sources are categorized as hardwoods (a.k.a. deciduous, angiosperms) and softwoods (a.k.a. conifers, gymnosperms). Softwoods commonly have fiber lengths of three to five millimeters, while hardwoods have fiber lengths of just one to two millimeters. Fiber width (diameter) may average 30 to 40 micrometers for softwoods and 10 to 20 micrometers for

hardwoods. Fiber length and width within a single tree will vary based on location and whether they come from springwood (earlywood) or summerwood (latewood). While softwood fibers give paper more tear strength, hardwoods form smoother surfaces. The pulp fiber mixture, called the furnish, is carefully controlled to optimize paper properties.

The major chemical components of wood are cellulose, hemicellulose, and lignin. Cellulose, a long-chain polymer of glucose, makes up about half of the wood fiber. It is the basic structural material of the cell and the site of hydrogen bonding between paper fibers. Hemicellulose can be broken down more easily and has a less ordered structure than cellulose. Lignin is an amorphous noncarbohydrate with a high molecular weight and is responsible for the brown color of unbleached pulps and yellowing of papers made from mechanical pulps. Softwoods tend to have more lignin than hemicellulose, while the reverse is true for hardwoods.

Pulping is the process of liberating fibers from the source material, usually wood and sometimes plant. This can be done mechanically, chemically, or with a combination of both (semichemical). These processes leave their marks on the texture and chemistry of paper fibers, and can be used to determine their origin by microscopic study. Most chemical pulping is now accomplished by the kraft process, using alkaline pulping liquors. Sulfite pulping, an acid process previously dominant, now accounts for only a small fraction of chemical pulps. Of the mechanical pulping methods, stone groundwood uses mechanical grinding action alone and causes significant fragmentation of the fibers. Other methods of mechanical pulping presoften the lignin, allowing fiber liberation with less damage and mechanical energy.

Bonding agents (dry-strength additives) may be added to pulps to improve bonding. Sizing agents impart water resistance to paper. Wet-strength additives improve paper performance in wet applications. Other additives are used for retention of fiber fines and fillers.

Fines created in refining can improve bond strength and smoothness. Fillers include mineral pigments added to improve smoothness and optical properties, and reduce the amount of fiber needed.

Recycled (also called secondary) fiber is becoming a large component of many paper and board products. Secondary fibers do not bond as well as virgin cellulose fibers. They do not rewet to the same degree and the fiber-fiber bond strength is weaker (1). Other problems with recycled fiber include the removal of stickies from adhesive-backed labels and envelopes.

2.2 Paper Structure

Paper structure and properties vary in three dimensions. Machine-made paper has a machine direction (MD) parallel to the movement of the paper on the paper machine. The perpendicular cross machine direction is referred to as CD. The long axis of fibers in paper has a tendency to orient near MD. Papermakers try to minimize variability of paper properties in the CD and carefully control the profile in the thickness or z-direction (2). Most papers have somewhat distinct felt and wire sides, the wire side being the underside of the paper facing the drainage wire after the headbox. Variations in the z-direction can appear as an uneven distribution of filler, fines, binders, and porosity. With an increase in twin-wire formers and improved machine operation and retention aids, the side differences are no longer as distinct.

Paper is an engineered material comprised of a network of fibers bonded at their contact points (3). It is instructive to look at paper on a variety of scales in order to understand its structure and performance. Electron microscopy allows such an examination. Figure 3 illustrates this in cartoon form. The drawing does not depict fillers and other additives that may be present in commercial papers.

Paper strength comes from both individual fiber, fiber bonding, and network properties. In Figure 3 the fiber *network* has lines showing the lateral compression of fibers at their contact/bonding points as a result of drying and shrinkage. The fine hairs on the surfaces of fibers represent external fibrillation, the result of fibrils being partially freed from the fiber layers during refining. Fibrillation and fines liberation enhance fiber-to-fiber bonding.

In Figure 3 the *fiber* window depicts the wood fiber substructure. The open core of the fiber is called the lumen. The bulk of the cell wall is the thick S2 layer. The orientation of microfibrils (microfibril angle θ) differs in the S3, S2, and S1 layers. Lignin content increases towards the outer layers and is highest in the middle lamella between fibers in the wood. The *fibrils* window shows the packing of fibrils and the concentration of lignin and hemicellulose between them. Finally, the individual fibrils and cellulose structure are depicted.

Refining the pulp promotes the fibrillation and collapse of fibers from their initially un-collapsed cross sections. Figure 4 shows an unrefined softwood kraft pulp, illustrating partial collapse of the fibers. Figure 5 shows the same pulp after 50 minutes of beating. Note the flattening of the fibers, close fiber-fiber contact, and microfibril bonding. When fibers do not fully collapse, the structure remains more open with higher porosity and bulk.

3. APPROACHES TO PROBLEM SOLVING

3.1 Transmission Electron Microscopy

The applications of TEM in the paper industry have been varied but generally fundamental in nature. The most common have been to document changes in the distribution of lignin in fibers due to pulp treatment. The distribution of lignin during ozone bleaching has been traced using TEM by Darabie et al. (4). Others have looked at the structure of cellulose in fibrils (5). Nanko

et al. (6) used TEM to demonstrate the microstructural effects of refining and the nature of crosslinking during interfiber bonding. One unusual application has been the measurement of mass distribution (known as paper formation) by using the electron beam in the TEM as a beta source and placing the sample in close contact with the recording film (7). The size and distribution of latex binders for coating pigments have been characterized by TEM (8). TEM can be used to study the size and shape of fine pigments, along with the functional interaction of additives with wood fiber.

A review of SEM and TEM preparation for, and the results of, wood fiber imaging was made by Duchesne and Daniel (9). The evolution from replica preparation to dominantly embedding and microtomy for TEM is described. External and internal characteristics of pulp fibers can be altered as much by EM preparation methods as by pulp treatment.

3.2 Scanning Electron Microscopy

Wood ultrastructure has been studied by electron microscopy to understand the origin of different cell types in hardwood and softwood. Scanning electron microscopy (SEM) beautifully reveals the three-dimensional morphology of wood. Butterfield and Meylan (10) published an excellent album of SEM photomicrographs of wood structures. SEM has also been used to document characteristics of individual cells to identify wood species in pulp and paper (11) and to illustrate the nature of papermaking materials (12).

The large depth of focus of SEM is extremely valuable in the study of wood and paper, both of which are highly textured at relatively low magnifications. Using stereo pairs, three-dimensional views can be obtained. Gregersen et al. (13) generated quantitative depth profiles

from SEM stereo pairs to measure the effectiveness of ink transfer to newsprint. Such measurements could potentially be used for surface roughness calculations.

Cellulose-based fibers are easily damaged using accelerating voltage and beam current settings that are common for inorganic materials (e.g., 15 kV, 2 nA), especially if magnifications over 5000 times are used. Epoxy-mounted cross sections are especially susceptible to degradation in the beam. Accelerating voltages of 5 to 10 kV for secondary electron imaging and 10 to 12 kV for backscattered electron imaging are usually safe and effective. Lanthanum hexaboride sources and field emission scanning electron microscopes (FESEM) allow higher resolution and signal strength at lower accelerating voltages than conventional SEMs. Duchesne and Daniel (9) demonstrated significant improvement in visualizing fibril structure detail using FESEM.

Low-vacuum and variable-pressure SEMs, including the environmental SEM (ESEM®), are growing in popularity in the paper industry because paper and coatings can be examined without reducing their moisture content. Tensile strengths of individual virgin and recycled fibers have been measured in an ESEM while imaging the failures (14). The degradation of paper surface properties during wetting were studied by moistening within the ESEM and found to be related to differential fiber swelling (15, 16). ESEM/EDS was one means used to evaluate the nonuniform coating pore structure responsible for backtrap mottle on offset printed sheets (17). Ink particles in recycled mixed office waste coming from laser printers and photocopiers have been measured with ESEM before and after deinking trials (18). The progression of solid-liquid-gas reactions can be videotaped with an ESEM.

3.3 Sample Preparation

Unless the analysis will be performed with a low-vacuum or variable-pressure SEM, moisture in the paper should be minimized as a first step in EM sample preparation. Sometimes it is sufficient to air dry or oven dry the material, depending on the objective of the examination. If there is reason to suspect that air, vacuum, or oven drying will collapse or otherwise alter a structure of interest, then critical point drying or freeze drying may be used. Freeze drying is potentially more destructive due to ice crystal formation or cell-wall collapse. In critical point drying, water in the paper is replaced with a dehydrating solvent followed by a transitional solvent (usually carbon dioxide). The carbon dioxide is introduced as a liquid under pressure, then taken above its critical point, where it is ideally vented without a meniscus forming.

Paper samples can be mounted on an SEM planchet using conductive paint or tape. For analysis with a conventional SEM, a conductive coating is needed. Evaporated carbon or plasma-applied coatings of metal (e.g., gold, gold-palladium) are the most common. Metal coatings provide better signal strength and resolution, but can interfere with EDS analysis and BSE imaging. If the application requires magnifications approaching 50,000 times or more, chromium or osmium coatings can improve performance.

Carbon coating is normally preferred to metal coatings for BSE imaging to optimize compositional contrast in the sample by minimizing the shielding of primary and backscattered electrons. However, Helle and Johnson (19) found that a gold coating was superior to carbon for enhancing the BSE contrast of offset and flexographic ink characters when the gold thickness and accelerating voltage were optimized.

3.4 Cross-section Analysis

The power of SEM for paper analysis is demonstrated in the ability to visualize the z-dimension, or cross section, of paper and board. Multi-ply tissue and board products are designed to optimize properties like smoothness, strength, and bulk while minimizing cost. Many paperboard boxes have an outer coated layer for high-quality printing. High end inkjet papers sometimes contain exotic materials to enhance photo quality. The composition, uniformity, bonding, and porosity of layers may be characterized by SEM analysis of cross sections.

The thickness of a paper, board, or coating depends on the intended application. Papers are typically 60 to 100 μm thick and paperboards are over 200 μm thick. In thinner papers, there may be only a few fiber layers making up the thickness (caliper) of the sheet. Figure 6 shows the cross section of a lightweight coated paper that is thin (37 g/m^2 basis weight) and coated on both sides. The base sheet was precalendered, coated, and then calendered again. Note the flattening of the fibers and the smoothness of the coated surface.

Cross sections of paper and board can be prepared in any number of ways. The most basic method is a razor-blade cut. This is usually unsatisfactory because of the damage and distortion it causes. Some modified razor methods using sharper blades and various substrates are adequate for certain tasks. A simple razor method using a common superadhesive to stiffen the paper was described by Fujita and Harada (20). However, it is more common to freeze-fracture the paper or board. This is accomplished by immersing the sample and blade in liquid nitrogen until they are thermally equilibrated and applying pressure from the razor's edge. Alternatively, the paper can be snapped with two pairs of forceps.

Most cross-sectioning involves impregnation with an epoxy resin. If resin-embedding a wet pulp, several dehydration and resin exchange steps are needed. For examination in an SEM,

it is not necessary to cut a section, but simply to expose a clean face for imaging. The two most common methods of preparing a cross section are microtomy and polishing. Polishing has the advantage of accommodating several paper samples in a single mount with more than a 2 cm exposure of each. It also avoids the wrinkling and distortion problems of cutting large microtome sections.

The thickness of a paper as measured by embedding and cross sectioning will commonly be less than the caliper measured in a paper-testing laboratory (under a standard 50 kPa platen pressure). This is in part due to resin shrinkage, but a greater factor is the three-dimensional surface texture of paper and the nature of the platens used to measure caliper. If one thinks of the paper surface as having a texture like a carpet, the hard platen of the caliper gauge does not conform to the surface roughness on both sides of the paper. The use of soft caliper platens (21) yields a smaller thickness, but it will not likely be as small as the two-dimensional profile exposed in cross section. Dickson (22) found that the thicknesses of Spurr resin-embedded cross sections of a thermomechanical pulp, as measured by confocal laser scanning microscopy, were 4% lower than micrometer measurement for uncalendered sheets and 19.5% higher for calendered sheets. Therefore, caution is advised when reporting paper thickness from cross sections.

Paper polishing with modified metallographic polishing methods was described by Gibbon et al. (23). Paper samples may have to be taken through a dehydration sequence prior to resin exchange, depending on their moisture content. Usually 25 to 32 mm diameter molds are filled with low-viscosity epoxy after the edge-oriented samples are placed inside. One face is ground and polished using a sequence of finer abrasives. Water-based lubricants are avoided to prevent swelling. Instead, grinding can be done dry or with alcohol or oil-based lubricants.

Allem (24) measured the thickness variation of coatings on lightweight coated (LWC) papers using SEM image analysis from polished mounts. The author used backscattered electron (BSE) imaging because it provides compositional contrast for the mineral pigment against the fiber and mounting resin. Care must be taken to remove abrasive residue as it may look similar to mineral pigment. Williams and Drummond (25) took the polishing procedure one step further by etching back some epoxy from the block face after polishing. This provides surface relief within the paper and facilitates high-resolution secondary electron (SE) images of the fiber, pores, and coating (if present).

4. PAPER COMPONENTS

4.1 Fiber Structure and Chemistry

Staining with bromine gas or potassium permanganate solution will raise the atomic number contrast of pulp fibers by reaction with the lignin (26). Using BSE imaging in the SEM, wood fibers are strongly contrasted with epoxy resin. In polished sections or on paper surfaces, differences in lignin content due to pulping and bleaching procedures are reflected in differences in gray levels of the fibers. This technique cannot be used to quantitatively determine lignin content due to variations in stain reactivity with different pulps or layers within the fiber. It has proven valuable in the measurement of cell-wall thickness changes due to hydrocyclone separation of mechanical pulp (27).

When individual pulp fibers are oriented down axis, the physical and chemical effects of papermaking can be monitored by EM. Cisneros et al. (28) reported on cell-wall degradation in mechanical pulps using SEM and TEM. Bleached chemithermomechanical pulp (BCTMP) was treated with thioglycerol to evaluate its effect on the delaying brightness reversion (29), the

yellowing observed as papers made with mechanical pulps age. The authors used SEM/EDS to measure the distribution of sulfur in the cell wall as a consequence of this treatment.

4.2 Pigments in Fillers and Coatings

Pigment is the general term for fine-grained nonfibrous pulp additives whose functions may include adding opacity, smoothness, brightness, or color, and reducing the use of fiber. Pigments are used as paper fillers and as coating components. Their particle sizes range from about 0.10 to 10.0 μm, although they are rarely over 5 μm. Common mineral pigments are kaolin clay, calcium carbonate (calcite and aragonite), titanium dioxide (anatase and rutile), and talc. Plastic pigments, usually as styrene-based spheres, may be mixed with the inorganic pigments.

The size and shape of pigment as seen in the SEM provides valuable clues to their identification. Talc and kaolin tend to be platy. Coarsely ground natural calcium carbonate (GCC) particles have irregular edges. Precipitated calcium carbonate (PCC) is made in a variety of shapes, but usually has well formed crystals. Spherical plastic pigment has easily identifiable solid and hollow forms.

SEM/EDS is a quick way to evaluate the type and two-sidedness of filler distribution in a paper. It requires only air dried mounts of both sides of the sheet. Using BSE imaging, mineral filler stands out as bright particles against the lower atomic number fiber background. Image analysis can be used to derive a relative comparison of filler coverage on the two sheet sides.

A true mineral identification requires structural identification, such as by diffraction, optical refraction, or FTIR. Elemental fingerprints from EDS are, however, useful in making tentative identifications. The presence of aluminum and silicon with roughly equal x-ray intensities is indicative of kaolin. Magnesium, with aluminum, silicon, and sometimes iron,

suggests that talc is present. Calcium alone (or with carbon and oxygen for light element detectors) is typical of calcium carbonate, but could be confused with calcium oxalate. Titanium is strongly indicative of titanium dioxide. Figure 7 is an EDS spectrum of a paper containing calcium carbonate and kaolin.

A common procedure for quantitatively determining the amount of inorganic additives in pulp, paper, and board is to weigh the residue after ignition (ashing) at 525°C. EDS analysis of the ash is a quick way to identify elements in the filler and contaminants present above the limit of detection (approximately 0.1 weight percent). Ashing also has the benefit of concentrating the solids from a large paper sample into a small volume.

4.3 Functional Additives

Retention aids help prevent the loss or uneven distribution of pigments and fines during the papermaking process. Like the mineral pigments, some additives may have multiple functions. Starch can be a sizing agent (improving water resistance), a binder in coatings, and/or a bonding agent.

Electron microscopy aids in evaluating the performance and improving the design of paper coatings and fillers. This may involve the characterization of the pigments and other components prior to mixing, or examination of the filler and coatings in the finished paper or paperboard. Nonpigment additives for paper include retention aids, sizing agents, wet-strength agents, and dry-strength agents. In addition to pigment, coating formulations require a binder and other components, like dispersants and plasticizers.

4.4 Paper and Board Coatings

Surface porosity of coated papers prepared for offset printing were evaluated by SEM (30). The objective was to determine factors affecting print gloss. When compared to measurements of pore volume and pore throat size by mercury porosimetry, the authors concluded that the surface pore structures differed from the interior of the coating. The relatively small surface pores impeded the absorption of ink and resulted in the development of higher print gloss.

Migration of the binder in coatings and its subsequent nonuniform distribution can result in poor coating properties and printing problems. In order to determine the location of latex binder in coatings, coated papers have been stained with osmium tetroxide and examined in the SEM. In an investigation of print picking, Smith et al. (31) used BSE imaging of cross sections to find the brighter (osmium-stained) latex. Kohno and Hamada (32) correlated latex distribution with basis weight variation over the surface of a coated paper that had been osmium stained. The authors used EDS to map the osmium Lα x-ray intensity across the surface. Cryogenic SEM, in which the sample is maintained in a frozen state, has been a powerful tool in the evaluation of wet coatings during consolidation. The behavior of latex binder and its interaction with mineral pigment has been documented with cryo-SEM (33, 34). With the availability of the ESEM® for wet samples, however, the complexity of cryo-SEM operation may limit its future application.

5. CONCLUSIONS

The end-user may not appreciate the engineering involved in the design and manufacture of paper and board for diverse applications in printing, packaging, and absorbent products. Electron microscopy reveals surface and internal characteristics of interest to those concerned with product development, product quality, and fundamental properties of paper and its

components. Scanning electron microscopy in its various forms has become an essential tool for paper characterization. Transmission electron microscopy enables fine structure resolution and visualization of the interaction of paper constituents.

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FIGURE LEGEND/CAPTIONS

Figure 1 Facial tissue showing open structure, high relief at the surface. SEM. Scale bar 10 μm.

Figure 2 Xerographic paper with mineral filler. SEM. Scale bar 10 μm.

Figure 3 Illustration of paper structure at several scales, from the network of fibers to the cellulose molecule. (Courtesy of G.A. Baum)

Figure 4 Sheet formed from unbeaten softwood kraft pulp. Low degree of fiber-fiber bonding. SEM. Scale bar 150 μm. (IPST archives)

Figure 5 Sheet from same pulp as in Figure 4, after 50 minutes of beating. Shows fiber collapse and high bond area. SEM. Scale bar 150 μm. (IPST archives)

Figure 6 Polished and etched cross section of lightweight coated paper. SEM. Scale bar 20 μm. (Photo by G. Maghiari)

Figure 7 EDS elemental spectrum of paper containing calcium carbonate and kaolin. Beryllium window acquisition mode.

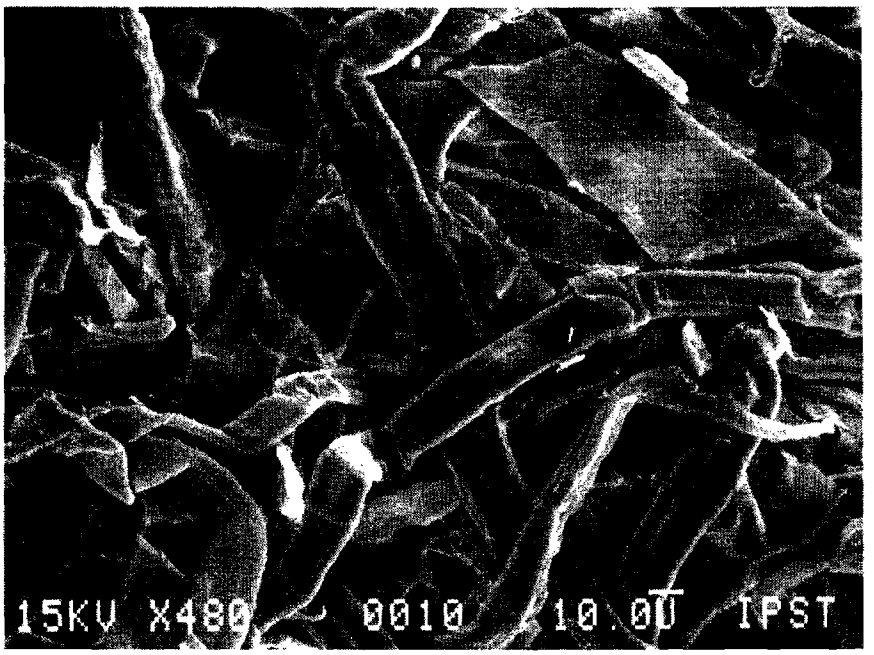

Figure 1

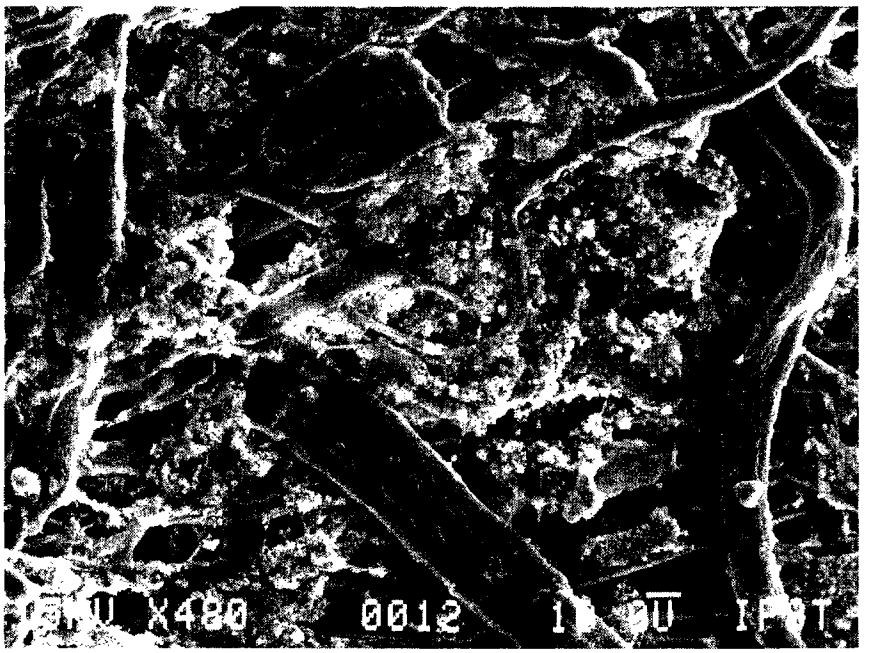

Figure 2

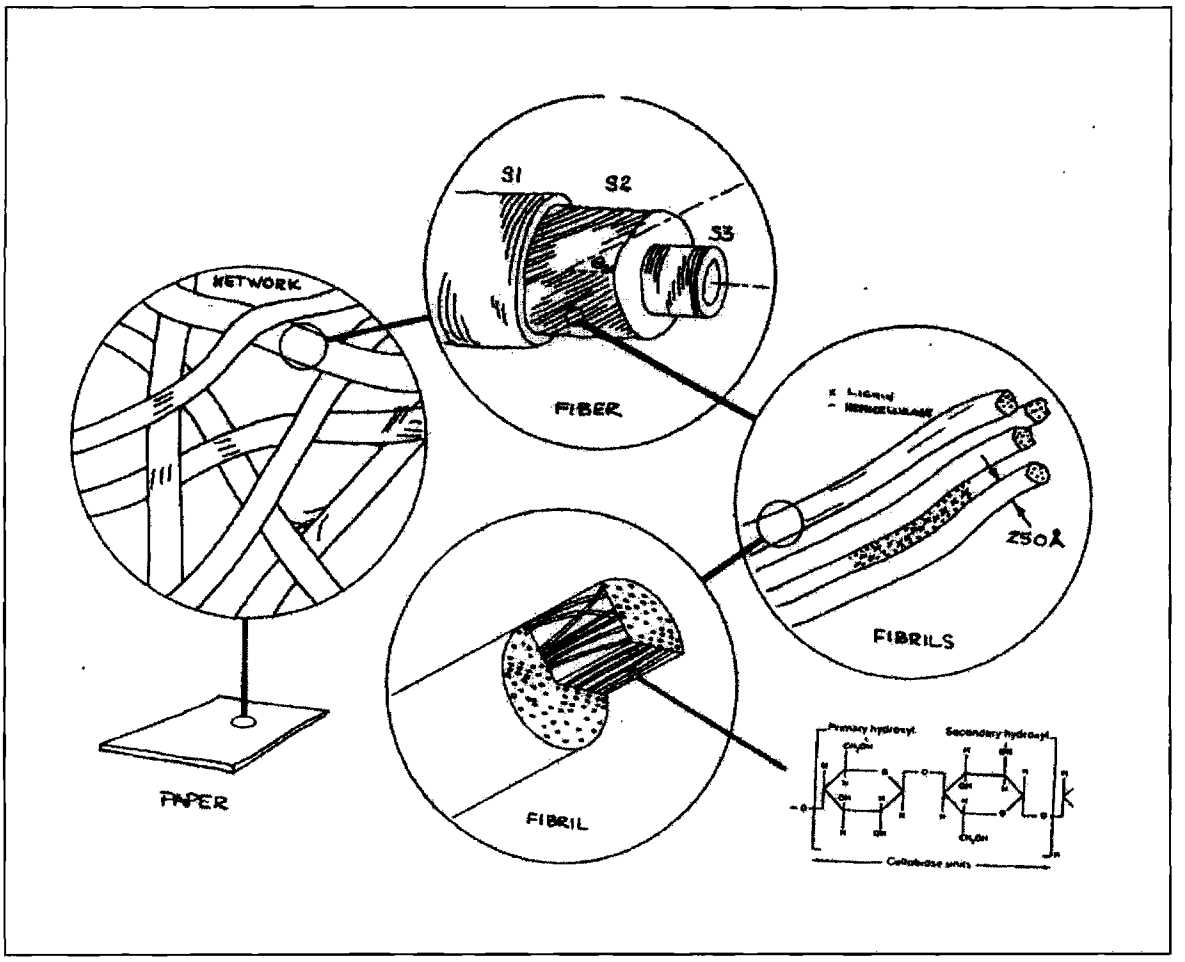

Figure 3

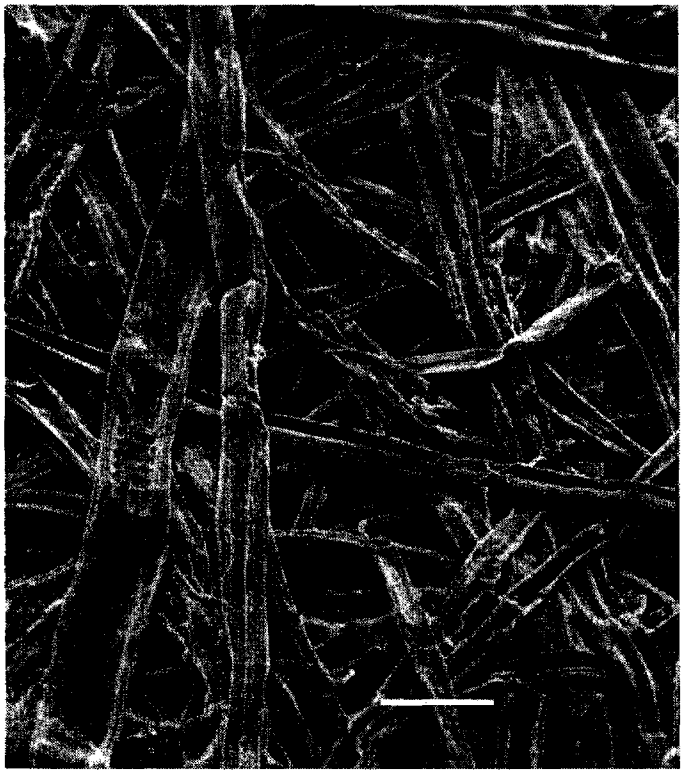

Figure 4

Figure 5

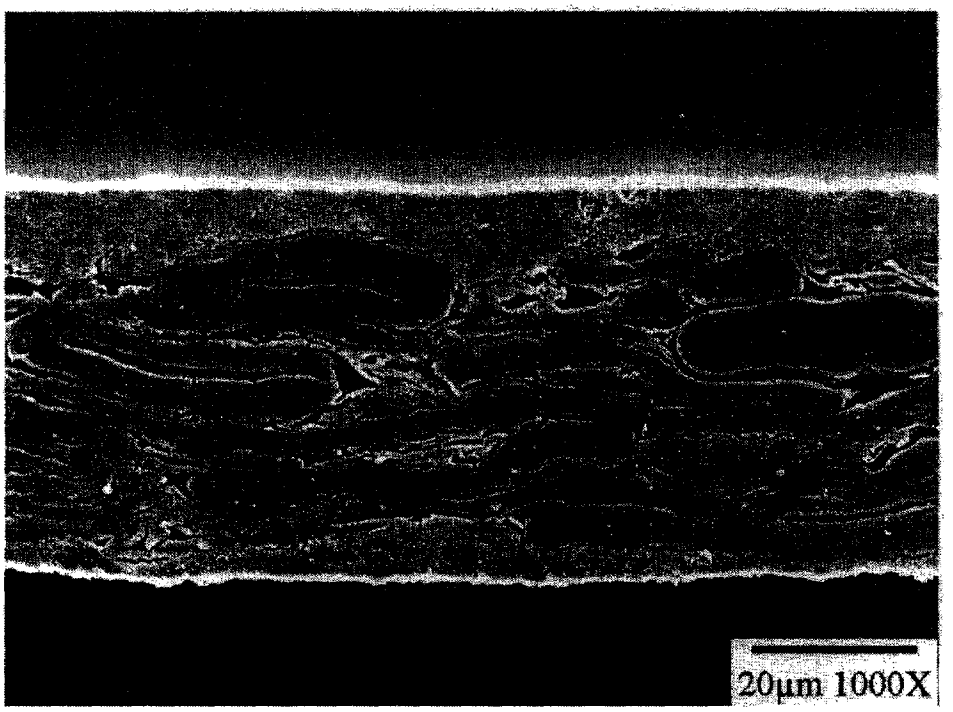

Figure 6

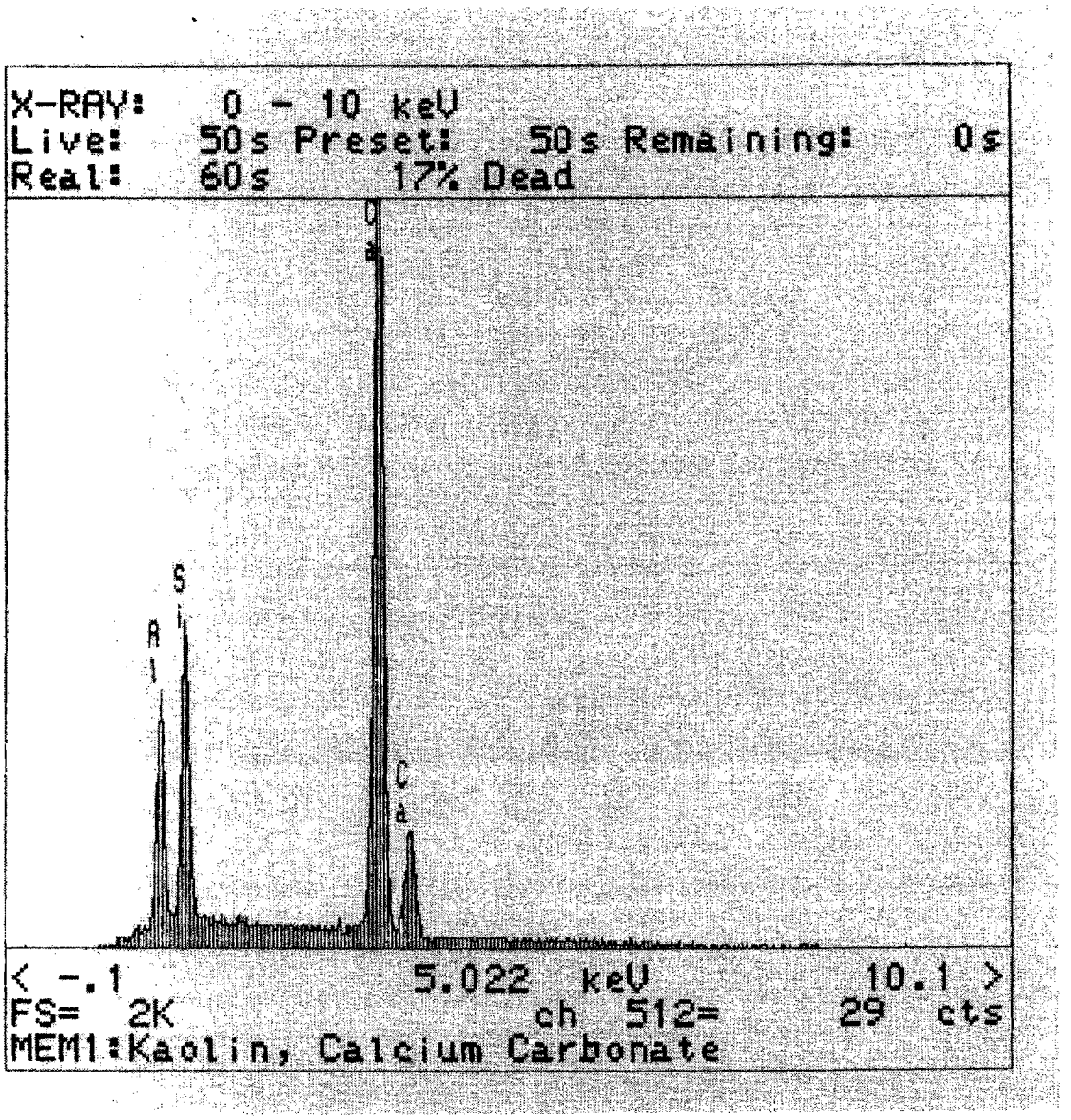
X-RAY: 0 - 10 keV
Live: 50s Preset: 50s Remaining: 0s
Real: 60s 17% Dead
Ca
Si
Al
C
a
< -.1
5.022 keV
10.1 >
FS= 2K
ch 512= 29 cts
MEM1:Kaolin, Calcium Carbonate

Figure 7